SAMPLING PROTOCOL

DETERMINATION OF POLYCHLORINATED DIBENZO-P-DIOXINS AND DIBENZOFURANS IN REDACTED

Study Number
AG000008

Test Substance:

Redacted (CAS # Redacted)

Prepared For:

Albaugh, Inc.
121 NE 18th Street
Ankeny, IA 50021

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REDACTED

APPROVED:

Ron Collins — 3-16-00
Ron Collins
Study Director
Albaugh, Inc.
Date

James Kahnk — 3-16-00
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DETERMINATION OF POLYCHLORINATED DIBENZO-P-DIOXINS AND DIBENZOFURANS IN REDACTED

1.0. Principals

1.1 Sponsor: Albaugh, Inc.

121 NE 18th Street
Ankeny, IA 50021
Phone: (515) 964-9444
Fax: (515) 964-7813

Study Director - Ron Collins
Phone: (816) 238-3377
Fax: (816) 238-3938

1.2 Sampling Facility: Blackman Uhler Chemical
Division of Synalloy Corporation
Augusta Chemical Plant
1010 Glass Factory Ave.
Augusta, GA 30901

2.0 Proposed Experimental Start Date July 1, 2000

3.0 Proposed Experimental Termination Date August 1, 2000

4.0 Data Requirements

U.S. Environmental Protection Agency (EPA) Toxic Substance Control Act (TSCA); Good Laboratory Practice Standard, 40 CFR Part 792; and the Dioxin/Furan Test Rule, 40 CFR Part 766. Under these provisions, this study is defined as a physical and chemical characterization study designed to determine certain physical and chemical characteristics of the test substance redacted (see 792.135(b); (54 FR 34094)).

5.0 Objective

The purpose of this study is to obtain samples of redacted that are typical of the material imported by Albaugh, Inc. for manufacturing. Samples of redacted manufactured by Anupam Rasayan (1661 Suthar Street, Nanpura, Surat 395001, India) will be sampled at Albaugh, Inc.'s toll manufacturing plant; Blackman Uhler Chemical (1010 Glass Factory Ave., Augusta, GA 30903).

The samples will be shipped to Battelle where the samples will be analyzed for PCDD/PCDF under the direction of Dr. Mark Bauer. Analyses will be conducted according to the "Analysis Protocol for Determination of Polychlorinated Dibenzo-p-dioxins and Dibenzofurans in Redacted". The analysis results will be used to meet regulatory requirements promulgated by the U.S. Environmental Protection Agency; Dioxin/Furan Test Rule, 40 CFR 766.

6.0 Test Substance and Test System

6.1 Test and Substance

The test substance for this study is listed below. Albaugh, Inc. will maintain a certificate of analysis and other chemical and physical characterization as required by Good Laboratory Practice Standards. Albaugh, Inc. will also maintain the stability, storage conditions, and will maintain the documentation for the synthesis and characterization of the test substances.

Common Name:	redacted
Chemical Name:	redacted
CAS Number:	redacted
Empirical Formula:	redacted
Molecular Weight:	redacted
Structure	redacted

6.2 Test System

The test systems for this study will be redacted.

6.3 Justification for Selection of the Test System

The test system for this study is the same as the test substance upon which all analyses will be conducted. The test system chosen is the actual substance to which the Dioxin/furan Test Rule applies.

6.4 Identification of the Test System

All test system samples will be labeled with a unique sample identification code. A unique identification code will be used so that no sample numbers are repeated.

Each sample will be identified by the following code using information taken from the barrel label's batch number and drum number:

BB-MM-NNN-BB-D

Where: BB = batch number information

MM = manufacturer information in batch number

NNN = compound name information in batch number

BB = batch number information

D = randomly selected drum number

For example, "12-AR-RED-34-3" represents the 3rd drum from batch 12-AR-RED-34 of redacted manufactured by Anupam Rasayan.

7.0 Experimental Design

7.1 Background

The code of Federal Regulations, Title 40, parts 707 and 766 (40 CFR 707 and 766) presents a rule for testing whether certain specified chemical substances may be contaminated with halogenated dibenzo-p-dioxins and dibenzofurans (The Dioxin/Furan Test Rule). This protocol describes procedures for sampling

redacted imported by Albaugh, Inc. for use in the production of their commercial product. The samples will be analyzed by Battelle for the determination of 2,3,7,8-substituted tetra- through hepta- chlorinated dibenzo-p-dioxins and dibenzofurans following an analysis protocol, which is presented as a separate document.

7.2 Normal Production Process

The redacted is manufactured by Anupam Rasayan, in India. Redacted is produced in a batch process. Benzene is chlorinated to 1,4-dichlorobenze and then nitrated to form 2,5-dichloronitrobenzene. This product is redacted to form redacted which is then converted to a redacted before it is redacted to form redacted.

The finished product is a liquid material and is packaged into closed-head, galvanized drums for shipment. Once in the drums, redacted solidifies.

7.3 Sampling Design

By means of random sampling, a minimum of seven (7) samples of redacted will be taken. Each sample will be taken from a different manufacturing batch.

7.3.1 Definition of Product Cycle

The production cycle is one batch per day. Each batch contains approximately 1250 kg of finished product. Five 250 kg drums are filled per batch.

7.3.2 Sampling Site

Sampling will take place after redacted is imported into the United States. It is not possible to control sampling and insure GLP compliance at the manufacturing plant in India. Sampling after import, at the end use site, also ensures that the samples collected are representative of the material used by Albaugh, Inc. Samples will be collected at Albaugh's toll manufacturing facility, Blackman Uhler Chemical, 1010 Glass Factory Avenue, Augusta GA 30903.

7.3.3 Selection of Samples

The redacted is imported in container load quantities. Each container holds 64 drums which represents more than 12 batches. A minimum of 7 different batches will be sampled. The drum sampled from each batch

will be randomly selected by using a random number generator. A programmable calculator or computer program will be used to generate random numbers between 1 and 5. For example, Microsoft Excel could be programmed with the following formula:

=ROUNDUP(RAND()*5,0)

An example completed random number selection is shown in the table below.

Batch #	Drum #
1	2
2	4
3	5
4	1
5	3
6	2
7	4

7.3.4 Sampling Process

The sampling procedures are those used for regular process quality control. Personnel collecting samples will wear appropriate protective clothing and follow all appropriate safety procedures when working with redacted.

The selected drum will be placed in a steam cabinet to re-liquefy the material. The drum will be opened and stirred to insure the contents are homogeneous. A sampling probe will be inserted into the middle of the drum to withdraw at least 100 g of redacted. The sample will be split into two 50-g sub-samples by pouring approximately half of the withdrawn sample into two clean, pre-weighed, properly labeled containers. One sub-sample will be labeled as the analytical sample, and the other labeled as a retain sample.

7.3.5 Sample Storage and Shipment

Sample container caps will be sealed with tape and the samples stored at ambient temperature.

The analytical sub-samples will be packaged for shipment to the analytical laboratory. The containers will be placed inside a secondary container (with any appropriate padding) to ensure the integrity of the other samples should a sample container break or leak during shipping. The samples will be shipped at ambient temperature using standard DOT shipping containers and following all relevant DOT shipping regulations.

The samples will be sent via an overnight carrier to:

Dr. Mark Bauer
Battelle
505 King Avenue
Columbus, OH 43201
(614) 424-3913

7.4 Documentation of Sampling

Sample data is recorded in sample labels, the Sample Data Form, the Chain-of Custody Form, and a sampling notebook.

7.4.1 Sample Labels

All sample containers will be labeled as follows:

Redacted
Analytical Sub-sample (or Retain Sub-sample)
Identification Number: _____
Sampling Date: _____
Sampling Time: _____
Weight (net): _____
Initials:
Store at room temperature

7.4.2 Sampling Data Form

A sampling data form will be used to record information about the samples. An outline of the sampling data form is as follows:

REDACTED SAMPLE COLLECTION DATA FORM

Date	Time	Sample ID	Container Weight	Sample + Container Weight	Sample Weight	Sampler's Initials

7.4.3 Chain-of-Custody Form

A properly completed chain-of-custody form, containing a complete record of sample disposition will be used to trace the samples. The chain-of-custody form will accompany samples during all sample shipments and/or transfers of custody. An example Chain-of-Custody Form is attached in Appendix A.

7.4.4 Sampling Record Book

In addition to the forms above, a complete record of sampling events and data will be maintained in a sampling record book or notebook. Data to be entered in this notebook includes: date, time, name of sampler, sample ID numbers, product batch or other drum label information, as well as any comments pertaining to the sampling. The notebook will also include a brief description of the sampling process and equipment used. A complete record of sample disposition, including date of shipments, mode of shipment, shipping tracking numbers, and address of receiver will be maintained in the notebook. Any deviations from written procedures, as well as any corrective actions taken on equipment will be recorded.

7.5 Corrective Action

Each sample is split into two duplicate sub-samples one of which is retained for replacement if needed. In addition, samples in excess of the minimum required may be taken such that a compromised sample may be replaced.

8.0 Control of Experimental Bias

The experimental design incorporates random sampling to control for experimental bias.

9.0 Records to be Maintained

All raw data and written records concerning the study will be part of notebooks established for the study. The study records will include, but not necessarily be limited to, the following:

1. This protocol, and any protocol amendments or deviations;
2. Test substance identification records, any characterization records supplied by the test substance manufacturer, and shipping and receipt records;
3. A full description of sampling conducted, including a description of the any equipment used;
4. Study raw data including:
   - Sample Collection Forms
   - Random number generation data
5. The final report and any amendments thereto.

10.0 Report

A final report detailing the sampling will be prepared by the Study Director. The final report will include a signed compliance statement attesting to whether the sampling was conducted in accordance with TSCA Good Laboratory Practice Standards.

The sampling report will be included, in it's entirety, as an appendix to the analytical analysis report.

11.0 Archive Statement

The study notebook(s), raw data, and other study records will be stored by Battelle, Columbus, Ohio after the approval of the final report. A copy of the final report will be archived with the study records after all approvals have been obtained.

All retained test substance will be maintained by Albaugh, Inc.

APPENDIX A

EXAMPLE FORMS

CHAIN-OF-CUSTODY FORM

Study Number: _____

Item Number	Sample Description	Sample I.D.
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Relinquished By: _____ Time/Date: _____ Received By: _____ Time/Date: _____

Relinquished By: _____ Time/Date: _____ Received By: _____ Time/Date: _____

Relinquished By: _____ Time/Date: _____ Received By: _____ Time/Date: _____

Relinquished By: _____ Time/Date: _____ Received By: _____ Time/Date: _____

Relinquished By: _____ Time/Date: _____ Received By: _____ Time/Date: _____

Relinquished By: _____ Time/Date: _____ Received By: _____ Time/Date: _____

Distribution: Original – Accompany Shipment
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